

### C. Remarks

This amendment is responsive to the office action dated November 20, 2007. In the office action, the claims were rejected under 35 U.S.C. 102/103 as being unpatentable over Armand and/or Chiang. A request for a one month extension of time for response is also included herewith. For the reasons set forth below it is submitted that the claims, as amended, are clearly patentable over the art of record. If any fees are required by this amendment, including the extension of time fee, please charge Deposit Account No. 502874.

Regarding the 35 USC.112 Rejection, claim 3 has been amended to correct the indefiniteness in the claim by deleting the parenthetical phrase "(aggregate)" noted in the office action.

This amendment is enterable under Rule 116 since it clarifies the claim language, raises no new issues and places the case in condition for allowance.

### The Invention

Claim 1 as previously amended reads as follows:

1. A method of producing olivine type nanostructured lithium metal phosphate of the formula,  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$ , where  $1 \leq x \leq 0.1$  and M is a metal cation and having an olivine structure, comprising the following steps:
  - (a) dispersing iron dextran nanoparticles in a liquid solution,
  - (b) at least one of the steps of :1) dissolving a metal salt in the liquid solution and 2) dispersing metal oxide nanoparticles in the liquid solution,

- (c) dissolving a phosphate ion containing chemical precursor in the liquid solution,
- (d) dissolving a lithium containing salt in the liquid solution,
- (e) heating the solution to promote the precipitation of  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  precursor material, and
- (f) evaporating the liquid and calcining the  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  precursor material for removing volatiles, and
- (g) annealing the  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  precursor material to form the olivine type nanostructured  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  compound.

#### The Interview

The applicants and their attorney wish to express their appreciation to the Examiner and his supervisor for the interview had at the USPTO on January 30, 2008 at which the present case and copending US Application S.N. 11/165,926 which is a CIP of the present application.

Based on the arguments presented at the interview the CIP application was allowed with the examiner citing certain art : A US patent to Lawrence (US No. 5,624,668) and a paper by Lawrence: "Development and Comparison of Iron Dextran Products"; PDA J. of Pharm Sci. Technology; Vol 52, pages 190-197, 1998. *It is respectfully requested that the Examiner make this art of reference here as well.*

We set forth below the arguments raised at the interview.

### The prior art

The present invention teaches a method for producing nanostructured  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  material where  $0.1 \leq x \leq 1$  and M is a metal cation. The present invention teaches the production of nanostructured olivine type  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  materials using nanoparticles of a unique iron-precursor, *iron-dextran powder*. The premise of adding nanoparticles of iron-dextran in an aqueous solution, along with the precursors of lithium, phosphate and metal cation (M) is that iron-dextran nanoparticles, which form a stable dispersion in water, will act as the *nucleation sites* to form nanophase  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  precursor material, which on annealing forms nanostructured  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  which is very suitable for use as a cathode material with an olivine structure. Nanophase  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  precursor material precipitates out from an aqueous solution. The solution is prepared by dissolving lithium and phosphate salts, by dispersing iron-dextran nanoparticles and either by dissolving a metal salt or dispersing metal oxide nanoparticles in water. This aqueous solution is heated at a temperature  $\leq 100^\circ\text{C}$  to promote the precipitation of nanophase  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  material, which is subsequently calcined and then annealed at an elevated temperature ( $> 400^\circ\text{C}$ ) in an inert atmosphere to produce nanostructured  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  material.

On the other hand, both Armand et al. (US 2004/0086445) and Chiang et al., adopted synthesis methods that are similar to a conventional solid-state method for producing a ceramic material wherein precursor materials only control the final composition, *but not the size of primary particles or crystallites* as taught in the present invention. In both cases, iron, phosphate and lithium precursors were mixed. Armand et

al. also included sugar, such as glucose and cellulose, as a source of carbon. However, in their case, sugar is not added to avoid the agglomeration of precursor material because the mixture was heated to a temperature high enough to decompose sugar molecules to produce carbon. Additionally, the starting raw materials were not nanoscale powders, and it was not taught or described that the size of raw materials will have a bearing on the size of the final product. Chiang et al. did not use a co-precipitation method. In their process, raw materials of different constituents were mixed by a ball mill in acetone. The solvent was evaporated to produce the dry mixture. Additionally, Chiang et al. did not make use of nanoparticles of precursor materials to produce nanostructured  $\text{LiFePO}_4$  or  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  materials.

However, the present patent application teaches a process for producing composite  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  powder using a unique iron-carbon precursor: iron-dextran. The iron-dextran compound (avg. particle size: 20- 30 nm) is composed of iron hydroxide nanoparticles that are chemically bounded with a dextran molecule with a typical molecular weight of 3000 - 5000. The iron-dextran compound supplies iron and is believed to provide nucleation sites to form pure phase  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  material. The nucleation sites provide a pre-configured nano sized precursor to assist in the growth of the final phase pure  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  particles.

The use of a iron-dextran pre-configured nano sized precursor as set forth in the claims as amended is not similar, nor obvious from, to previously disclosed or described methods, including Armand et al.'s patent application and Chiang. However, to produce

$\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  cathode materials with good electrochemical properties, in addition to composition, the particle (or crystallite) size of the material also need to be controlled. The present invention teaches the control of the particle (or crystallite) size by using the iron-dextran precursor (average particle size 20-30 nm), which has a specific core size (~ 3 nm) and a unique microstructure [see Lawrence US patent 5,624,668].

### Iron-Dextran

Regarding the issue of whether an iron-dextran complex was formed when Armand et al. synthesized  $\text{LiFePO}_4/\text{C}$  composite powder, iron-dextran is a core-shell nanoparticle with a "FeOOH" core. The core size is approximately 3 nm. Dextran chains/molecules form a complex with the FeOOH core and dextran chains string out from the FeOOH core in solution.

Since the core of Fe-dextran complex is FeOOH, the complex is formed by neutralizing an iron salt, such as iron chloride, with NaOH in the presence of dextran. According to US patent No. 2,820,740 (attached hereto and cited in the Lawrence patent), one route to synthesis of Fe-dextran involves the following process: partially depolymerized dextran is dissolved in water; appropriate amounts of NaOH and iron chloride are then dissolved in the dextran solution. This solution is heated to boil for 15 minutes. Undissolved solid materials are removed by a centrifuge, and the solution is dialyzed under running water for 24 hrs. The dialyzed solution is concentrated to obtain a

specific Fe concentration. The product is refiltered and sterilized in an autoclave. The pH of the final solution is 6.8.

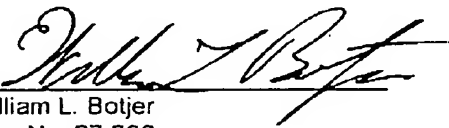
The above description, as well as the Lawrence patent and Lawrence paper demonstrate that forming Fe-dextran complex is an involved reaction, and simply dissolving an iron salt with a dextran compound will not result in the formation of Fe-dextran complex as the solution needs to be neutralized to form FeOOH core, which then complexes with dextran chains/molecules. Armand et al. (US 2004/0086445) produced  $\text{LiFePO}_4/\text{C}$  composite powders by mixing in solid-state or dissolving in solution the raw materials of iron, lithium, phosphate and carbon. The carbon precursor was either a sugar molecule or polysaccharide. Since they did not use the neutralization step, which is a prerequisite for forming iron-dextran complex, it can be concluded that iron-dextran complex never formed when Armand et al. synthesized  $\text{LiFePO}_4/\text{C}$  composite powder.

In summary, none of the art cited herein and in the CIP application provides a suggestion or motivation to use Iron dextran particles as a precursor in the synthesis of the present claims. This is because the Iron dextran compounds are used to treat iron deficiency of people and animals in the medical and veterinary fields. Furthermore, the method steps set forth in the prior art such as raising and lowering of Ph of the solution, the specific amounts of precursors and the specific heating and cooling of the solution and the neutralization of the solution thus the resulting properties and product would not be inherent to the mere mixing of an iron and polysaccharide components of the

prior art. Thus the invention set forth in the present claims would not be obvious to a person of ordinary skill in the arts of electro-conductive materials. It is respectfully submitted that the prior art cited herein and in the CIP application, when taken alone or in combination, does not teach the production of a nanostructured  $\text{LiFe}_x\text{M}_{1-x}\text{PO}_4$  material through the use of a iron-dextran nano sized precursor as recited in the present claims.

Thus, the claims as amended are clearly patentable over the art of record, and notice to that effect is earnestly solicited. If the Examiner has any questions regarding this matter, the Examiner is requested to telephone applicants' attorney at the numbers listed below prior to issuing an advisory action.

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